Phytate Extraction from Corn Ethanol Co-Products

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Background

In Minnesota, there are two main industrial corn ethanol production processes used: wet milling and dry-grind. While wet milling utilizes water soaking pretreatment for corn kernel, dry-grind process involves a physical processing of the kernels such as hammering. Dry-grind process generally costs less and is easier for other utilization of co-products. The co-products obtained from ethanol process are:

- Whole Stillage (WS) is the fermentation residue left from ethanol distillation that includes fibers, lipids, proteins and other minerals
- Thin Stillage (TS) contains most of the moisture content and is obtained from centrifuging WS
- Wet Distillers Grains is the solid from WS centrifugation
- Condensed Distillers with Solubles (CDS) is produced from drying TS
- Wet Distillers Grains with Solubles (WDGS) is produced from combining CDS with Wet Distillers Grains
- Dried Distillers Grains with Solubles (DDGS) the product obtained from drying WDGS and is often used as animal feed.

Problems:

- DDGS is used as animal feed for non-ruminant animals such as poultry and swine but they cannot digest phytate
- Phosphorus is excreted by animals and produces runoff with high phosphorus content when manure is used as fertilizer and cause environmental problem.

Values of phytate

- Phytate is a storage form of phosphorus in seed
- Phytate has good chelating properties and can be use as anti-oxidant in food
- It is also used in pharmaceutical and textile industry.

Methods of analysis

- HACH Phosphorus kit was used to measure the total phosphorus by the change of color using a spectrophotometer
- Megazyme Phytic Acid kit with phytase enzyme was used to test for phytate by determining the difference in the total and free phosphorus
- Ashing of samples was done by burning samples to ashes at high temperature and dissolving in HNO_3 and using the HACH kit to measure the total phosphorus in the samples.

Methods of extraction

Acid and water extraction

- Samples of Whole Stillage, Thin Stillage, Condensed Distillers Solubles, Wet Distillers Grains with Solubles and Dried Distillers Grains with Solubles were treated with 1 M HCl for acid extraction and distilled water for water extraction in order extract soluble phytate from the samples
- Extracted liquids are treated with $CaCl_2$ to precipitate phosphorus in salt form as calcium phytate
- Salt is washed and dried overnight and then dissolved in 1 M HCl for phosphorus analysis

Ion exchange

- Strong anion exchange resin was added to a column for ion exchange method
- Samples of CDS at different moisture content were added to the column to determine adsorption efficiency
- Resins are washed, filtered and desorbed with NaCl, HCl and NaOH as desorbing agents
- Eluents are precipitated with $CaCl_2$ and phosphorus content analyzed

Results

Sample	Moisture content (%)	Total P (mg/g)	Phytate P (mg/g)
WS	87.94±0.15	12.02±0.60	4.81±0.01
TS	95.71±0.13	23.57±1.12	10.11±0.66
WDGS	49.75±0.40	10.21±0.53	3.27±0.21
CDS	70.48±0.22	23.77±0.23	9.41±0.34
DDGS	14.33±0.39	10.59±0.20	4.50±0.15

Table 1. Characteristic of co-products

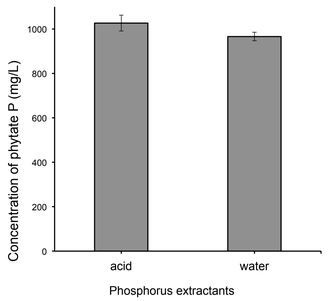

Figure 1. Acid and water extraction

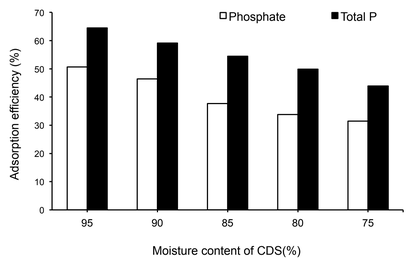

Figure 2. Moisture content and adsorption efficiency

Discussion

In Table 1, we can see that Thin Stillage with 95% moisture content has a higher concentration of phytate in comparison to CDS with 70% moisture content on a dry basis. Since about 70% of phosphorus after fermentation is present in TS, this means that phytate from animal feed can be greatly reduced by extracting phytate from TS alone. Also, in Figure 1, it can be observed that there is only a small difference in concentration of phytate in acid and water. This indicates that much of the phytate is readily dissolves in the liquid fraction of the samples. In Figure 2, we can see that the adsorption efficiency of the resin is highest at 95% moisture content of CDS, which is similar to that of the Thin Stillage. This shows that thin stillage can be used directly in extraction process, instead of adding water to achieve desired moisture content. In desorption process, it is found that NaCl at 1 M is most efficient, desorbing almost 90% of the absorbed phosphorus, producing a high concentration product.

Conclusion

In order to eliminate environmental problems and create additional values to corn ethanol co products, different methods of phytate extraction were studied. The moisture content of the samples has a significant influence on the phytate present. Thin stillage, with highest moisture content, yields the highest amount of phytate. Higher moisture content makes it more convenient for ion exchange resin to extract phosphorus. With NaCl as a desorbing agent, the end product yields a high concentration of phytate. Because most of the main components of the extraction method are found, the project can continue with an in-depth study on other parameters that can affect the extraction method such as time and temperature. It also creates an opportunity for developing the purification process for phytate.

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